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^{103}Rh-NMR spectroscopy of some hydridorhodiumbis(carbonyl)diphosphine compounds☆

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Dedicated to Professor A. Ceccon on the occasion of his 65th birthday.

Abstract

The ^{103}Rh chemical shifts of a series of hydridorhodiumbis(carbonyl)diphosphine compounds **1**–**17** and one phosphine–phosphite analogue **18**, containing chelating bidentate P-ligands, have been obtained by inverse HMQC detection sequences ^{1}H-{^{103}Rh} and ^{31}P-{^{103}Rh,^{1}H}. These active hydroformylation catalysts are stable only under pressure of H_2/CO (synthesis gas) and hence ^{1}H-, ^{31}P- and ^{103}Rh-NMR spectra have been recorded in a sapphire high-pressure NMR tube. The compounds $HRh(CO)_2(PP)$ exist as a mixture of equatorial–equatorial and equatorial–axial five-coordinate isomers for **1**–**17**, which are in a dynamic equilibrium that could not be brought into the slow exchange regime on the proton, the phosphorus or the rhodium NMR time scales. A correlation ($R = 0.980$) was found between $\delta(^{103}\mathrm{Rh})$ and the Hammett σ_p-constant of the *para*-substituents Y of the $-P(C_6H_4-Y\text{-}p)_2$ groups in the thixantphos ligand for the series of compounds **4**, **8**–**13** (lower $\delta(^{103}\mathrm{Rh})$ for electron withdrawing substituents). Correlation of $\delta(^{103}\mathrm{Rh})$ with Tolman basicity parameters (higher $\delta(^{103}\mathrm{Rh})$ with higher basicity) also gave a good fit ($R = 0.955$). The finding that correlations exist for this series between the ligand basicity, the ratio of equatorial–equatorial/equatorial–axial trigonal bipyramidal isomers and the $\delta(^{103}\mathrm{Rh})$ indicates that small structural and electronic changes in the ligands in the vicinity of transition metal nuclei have a small but significant influence on $\delta(^{103}\mathrm{Rh})$. This, together with other knowledge, may in the future serve to use Rh-NMR as an analytical tool in coordination chemistry. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: ^{103}Rh-NMR; Rhodium compounds; Phosphines; Hydrido compounds; Hammett relation

1. Introduction

^{103}Rh-NMR has long been considered difficult due to the low gyromagnetic ratio of that nucleus ($\Xi = 3.16$ MHz) [1]. The advent of inverse detected HMQC sequences [2–5] has greatly facilitated the acquisition of NMR data on such nuclei. Provided that a coupling $^{n}J(^{1}\mathrm{H},^{103}\mathrm{Rh})$ or $^{n}J(^{31}\mathrm{P},^{103}\mathrm{Rh})$ for coherence transfer is available, inverse 2D X, ^{103}Rh-NMR leads to a theoretical sensitivity enhancement with a factor of 360 for $X = {}^{31}P$ and 5630 for $X = {}^{1}H$, respectively [6].

Recent applications of ^{103}Rh-NMR comprise rhodium compounds containing ligands with e.g. phosphorus [7–10] and nitrogen [11,12] donor atoms. Most of the compounds studied thus far are relatively stable and isolable at ambient conditions. Correlations of transition metal chemical shifts with stability constants are known [13].

Transition metal NMR data on compounds of limited stability, such as intermediates and species generated in situ under pressure [14], are scarce. Access to such data could be of special interest for studies in homogeneous catalysis [15,16], e.g. ^{103}Rh-NMR data for homogeneous catalytic hydroformylation and ligand substitution reactions [17]. The gathering of such data is important in order to examine the influence of ligand environments on metal centers in cases which are

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☆ Publication of this contribution has been delayed because of patent priorities involving some of the authors.

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not amenable to spectroscopic study under normal conditions. They may in the future provide information concerning the composition and structure of similar species studied.

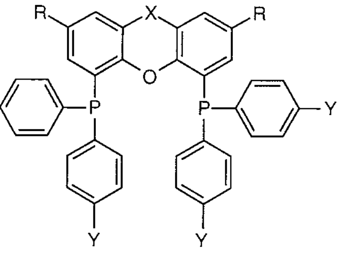
Here we present the results of a study on the ^{103}Rh chemical shift of hydridorhodiumbis(carbonyl)diphosphine compounds, which are stable only under elevated gas pressures of H_2/CO (synthesis gas). These compounds are active in the rhodium catalyzed hydroformylation of 1-octene and several of these give rise to high normal/branched ratios of the product aldehydes [18].

2. Results and discussion

The Compounds HRh$(CO)_2$(diphosphine) **1**–**17** and the analogous phosphine–phosphite complex **18** have been synthesized in situ [19] by reacting an appropriate amount of the diphosphine with Rh(acac)$(CO)_2$ in benzene-d_6 under syngas (CO:H_2, 1:1) pressure in a 5 mm titanium–sapphire high-pressure NMR assembly. This device consists of a 5 mm outer diameter, 3 mm inner diameter sapphire tube and a Ti-alloy pressure head, similar to the ones previously employed [20–23]. The diphosphines in the compounds **2**–**13** have been devised by Van Leeuwen [18,24]; the structures of the ligands are shown in Fig. 1.

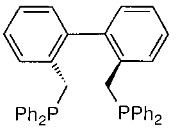
HRh$(CO)_2$(diphosphine) compounds **1** -**13**

diphosphine:



No.	X	Y	R
1	H,H	H	H
2	P-Ph	H	H
3	$SiMe_2$	H	H
4	S	H	Me
5	CMe_2	H	H
6	C=CMe_2	H	H
7	NH	H	H

No.	X	Y	R
8	S	CF_3	Me
9	S	Cl	Me
10	S	F	Me
11	S	Me	Me
12	S	OMe	Me
13	S	NMe_2	Me



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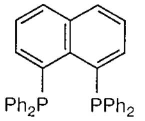

DPPN

Fig. 1. Compounds and ligands.

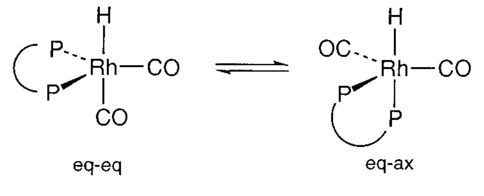

Fig. 2. Dynamic equilibrium between equatorial–equatorial (eq–eq) and equatorial–axial (eq–ax) five-coordinate Rh compounds.

Several of the HRh$(CO)_2$(diphosphine) compounds selected for this work have been used in studies of hydroformylation reactions, and their structure has been assessed by variable temperature NMR and IR spectroscopy [18 and this work]. They have to be prepared in situ and are not stable under ambient conditions. Their composition has been confirmed by ^{1}H{^{103}Rh}HMQC-NMR spectra, which show a triplet with typical ^1J(Rh,P) of 120–130 Hz. The variable temperature ^{31}P- and ^{1}H-NMR data together with IR data indicate that for **4** and **8**–**13** a mixture of five-coordinate trigonal bipyramidal isomers is obtained in which the diphosphine occupies equatorial–equatorial (eq–eq) and equatorial–axial (eq–ax) sites, the ratio of which depends on the specific diphosphine [18]. Similar behavior has been observed for **1**–**3** and **5**–**7** as well as for **14**–**17**. Compound **18** has eq–ax structure [25]. The dynamic equilibrium between eq–eq and eq–ax isomers for **1**–**17** (Fig. 2), which could not be brought into the slow exchange regime on the proton or the phosphorus NMR time scales, could generally not be frozen out on the rhodium NMR time scale either.

The ^{103}Rh-NMR data, obtained by ^{1}H-{^{103}Rh}HMQC as well as by ^{31}P-{^{103}Rh,^{1}H}HMQC sequences for almost all compounds, have been compiled in Table 1. Both methods gave the same results, confirming the correctness and accuracy of the presented data. Apart from the ^{1}H and ^{103}Rh chemical shifts, the coupling constants ^2J(H,P) and ^1J(P,Rh) can also be directly deduced from the ^{1}H-{^{103}Rh}HMQC spectra. Couplings ^1J(H,Rh) have been obtained from proton spectra. These data have also been collected in Table 1.

Nuclear magnetic shielding of transition metal nuclei is composed of a diamagnetic and a paramagnetic term. It is widely accepted that the former has almost no influence on the nuclear magnetic shielding; the nuclear shielding of transition metal nuclei like ^{103}Rh is mainly determined by the paramagnetic contribution σ_p to the overall shielding. It may according to the Ramsey equation, later developed by Griffith and Orgel [26,27], be described by changes in the average excitation energy, ΔE_{av}, the inverse cube of the d-orbital radius, $\langle r_d^{-3} \rangle$, and the angular imbalance of charge, $\Sigma\, Q_N$ (1).

$$\sigma = \sigma^d - \sigma^p = A - c(\Delta E_{av})^{-1} \langle r_d^{-3} \rangle \, \Sigma\, Q_N \quad (1)$$

The ^{103}Rh chemical shift of all compounds fall in the region expected for compounds with strong-field ligands such as carbonyl, hydride and (to a lesser extent), phosphines. They are observed at somewhat lower fre-

Table 1
NMR data for $(H)Rh(CO)_2(PP)$ [a]

Compound no.	P–P ligand	R	X	Y	Calc. bite angle (β_n)	$\delta(^{103}Rh)$ via ^1H	$\delta(^{103}Rh)$ via ^{31}P	J(H–Rh) (Hz)	J(H–P) (Hz)	J(P–Rh) (Hz)
1	DPEphos	H	–	H	101.6	−828.9	−828.6	11.0	45	124
2	Phenyl-P-xantphos	H	P–Ph	H	ca. 103	−828.2	−827.9	6.6	17	126
3	Sixantphos	H	Si–Me_2	H	106.2	−817.4	−817.1	8.1	22	124
4	Thixantphos	Me	S	H	106.4	−840.0	−840.1	6.6	15	128
5	Xantphos	H	C–Me_2	H	109.8	−800.8	−800.9	6.6	17	127
6	Isopropenyl-xantphos	H	C=CMe_2	H	ca. 110	−821.1	−821.0	6.6	13	128
7	*N*-Xantphos	H	NH	H	115–120	−785.3	−785.2	5.9	15	128
8	*p*-Trifluoromethyl-thixantphos	Me	S	CF_3	109.3	−850.9	−851.0	4.4	3.6	135
9	*p*-Chloro-thixantphos	Me	S	Cl	107.8	−840.7	−840.7	5.9	8.1	132
10	*p*-Fluoro-thixantphos	Me	S	F	106.6	−835.6	−835.7	6.6	11	131
11	*p*-Methyl-thixantphos	Me	S	Me	106.7	−831.5	−831.6	7.3	18	126
12	*p*-Methoxy-thixantphos	Me	S	Ome	106.9	−825.3	−825.4	7.3	21	125
13	*p*-Dimethylamino-thixantphos	Me	S	NMe_2	109.1	−814.3	−814.2	8.8	28	122
14	BISBI				112.6	−897.6	−897.6	ca. 2	7	149
15	DPPE [b]				84.5	−1073	n.d.	11.0	59.3	118
16	DPPP [b]				ca. 90	−954.9	n.d.	11.7	54	113
17	1,8-DPPN [c]				ca. 90	−953.6	n.d.	13.9	62	108
18	BINAPHOS [d]				n.d.	−969.6	n.d.	9.5	[d]	[d]

[a] At $T = 298$ K, solvent C_6D_6, unless otherwise specified. Ligands see Fig. 1.

[b] 1,2-Bis(diphenylphosphino)ethane; 1,3-bis(diphenylphosphino)propane; solvent THF-d_8.

[c] 1,8-Bis(diphenylphosphino)naphthalene.

[d] Nozaki/Takaya chiral phosphino–phosphite ligand [25]: $\delta(^1H)$ −8.92; $\delta(^{31}P_{eq})$ 27.6; $\delta(^{31}P_{ax})$ 185.4 ppm; $J(H–P_{eq})$ 22.7; $J(H–P_{ax})$ 158.8; J(H–Rh) 9.5; $J(P_{eq}–P_{ax})$ 38.6; $J(P_{eq}–Rh)$ 118.8; $J(P_{ax}–Rh)$ 182.7 Hz.

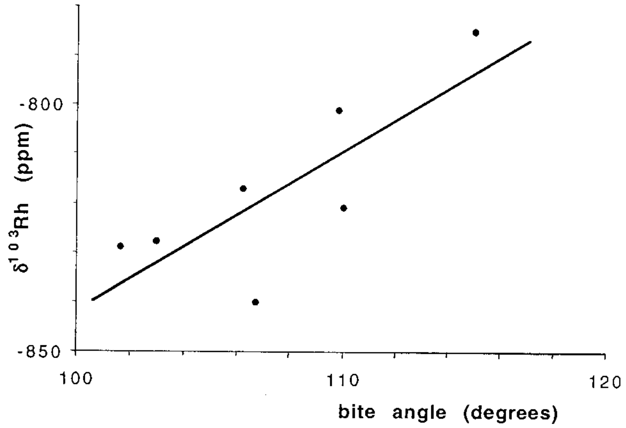

Fig. 3. The ^{103}Rh chemical shift of **4** and **8**–**13** as a function of the natural bite angle of the ligand.

quencies than, e.g. square pyramidal HRh(PMe$_3$)$_4$ at − 735 ppm [28], but at higher frequencies than for comparable four-coordinate compounds. This finding is in agreement with considerations of ligand field splitting, and hence with the relative magnitude of ΔE_{av} for the compounds in question. The main reason is, that for five-coordinate compounds the ligand field splitting will be slightly smaller than for four-coordinate square planar systems [29]. The energy of the $d_{x^2-y^2}$ orbital and hence ΔE_{av} is mainly determined by the ligands in the plane of five-coordinate compounds. The energy of the d_{z^2} orbital will be influenced to some extent by the apical ligand, but the latter does not, or only very slightly, contribute to the paramagnetic shift. It is therefor expected that the ^{103}Rh shifts for the compounds at hand will also be found near, but at somewhat higher frequencies compared to complexes such as [Rh(diphosphine)$_2$]$^+$. Indeed, the ^{103}Rh shift of [Rh(dppe)$_2$]$^+$ is observed at − 1172 ppm [8], which is significantly but not very much lower than those observed (Table 1) for the series **1**–**18**. The ^{103}Rh shift of compounds in this study bearing diphosphines similar to dppe, such as the sub-series **14**–**18**, are rather close to the observed shift for [Rh(dppe)$_2$]$^+$.

Remarkably, the chemical shift difference of 118 ppm between the complexes **15** and **16**, containing dppe and dppp, respectively, is almost the same as the difference found for square planar [Rh(nbd)(dppe)]$^+$ and (Rh(nbd)(dppp)]$^+$ (nbd = norbornadiene) [8]. This would suggest some rules of additivity of shifts in series of similar compounds, which has been noted previously [8,10] and may be the subject of a future investigation. Although the ligand in **17** is far more rigid than in **16**, the same ^{103}Rh chemical shift is observed for **16** and **17**. Both have a six-membered chelated diphosphine with about equal basicities and bite angles. The temperature dependence of the chemical shift amounts to 0.4–0.8 ppm K^{-1} (measured between − 100°C and + 20°C for **1**, **6**, **15**, **16**), which is within the normal range.

Compound **18** has close to eq–ax structure [25] with the phosphite P-atom *trans* to the hydride. Remarkably, the ^{103}Rh chemical shift of this compound is very close to that of **16** and **17**.

The ^{103}Rh chemical shifts of compounds **1**–**13** are all found in the relatively narrow range between − 785 and − 851 ppm, as would be expected for constitutionally similar compounds. As indicated above, the ^{103}Rh shows an average of eq–eq and eq–ax isomers. These are present in a ratio depending on the bidentate P-ligand [18]. First, we attempted to establish a correlation between the ^{103}Rh chemical shift and the natural bite angle of the ligands (Table 1), but this appeared unsuccessful, as is clearly seen from the graph (Fig. 3) and the low correlation coefficient $R = 0.793$. The calculated natural bite angles [24] of the free ligands were used. Accurate data on the actual coordination compounds are not available (no crystal structures are known of the compounds under study, which are unstable at ambient conditions).

It has been proposed that the transition metal shift parallels the variation of the bulk and basicity of the coordinated phosphine ligand. In a systematic study of Rh–phosphine compounds it has been shown that an increase in the basicity of the P-ligand, as well as of *para*-substituted pyridines, results in shifting $\delta(^{103}\text{Rh})$ to higher frequency [10]. A similar effect can be inferred from the $\delta(^{103}\text{Rh})$ data for the compounds Cp*Rh{C(O)–C$_6$H$_4$–Y-*p*}, i.e. an increase in $\delta(^{103}\text{Rh})$ with increasing electron donating ability of the *para*-substituent on the Ar group [30]. Recently it was found that differences in the distance of the donor atom (phosphorus or nitrogen) to Rh contributes far more to changes in the chemical shift than variations in the coordination angles [12,31]. Hence, ligand basicity and donor character may be more relevant in determining the paramagnetic shift. Such effects should then be reflected in the Tolman basicity parameters and Hammett σ_p-constants, at least for a series of similar compounds.

Indeed, a clear correlation ($R = 0.980$) was found between $\delta(^{103}\text{Rh})$ and the Hammett σ_p-constant [32,33] of the *para*-substituents Y of the –P(C$_6$H$_4$–Y-*p*)$_2$ groups in the thixantphos ligand in the series of compounds **4**, **8**–**13** (see Table 2 and Fig. 4). When omitting compound **4** the correlation coefficient was 0.991. Correlation of $\delta(^{103}\text{Rh})$ with the Tolman basicity parameters [34] also gave a good fit ($R = 0.955$).

Previously, a correlation of the basicity parameters of the same ligands with the ratio of eq–eq/eq–ax isomers of the HRh(CO)$_2$(thixanthphos) complexes **4**, **8**–**13**, as estimated from variable temperature proton NMR and IR data, has been observed [18]. The amount of eq–eq isomer in the equilibrium mixture correlates linearly with the Hammett σ_p-constants ($R = 0.987$). As the $\delta(^{103}\text{Rh})$ has been found to correlate with the Hammett

Table 2
The ^{103}Rh chemical shift of **4** and **8**–**13** as a function of the Hammett parameter σ_p

No.	Y-substituent	Hammett σ_p	δ ^{103}Rh
8	CF_3	0.54	−851
9	Cl	0.23	−841
10	F	0.06	−836
4	H	0.00	−840
11	Me	−0.17	−832
12	OMe	−0.27	−825
13	NMe_2	−0.83	−814

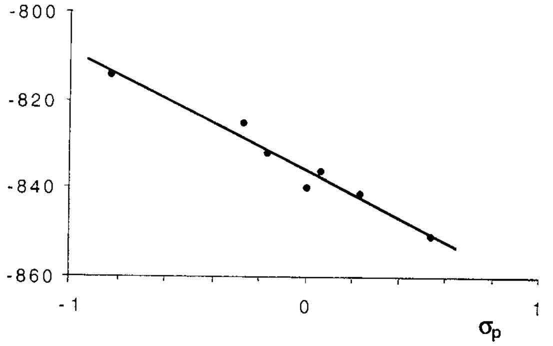

Fig. 4. The ^{103}Rh chemical shift of **4** and **8**–**13** as a function of the Hammett parameter σ_p. $\delta(\text{Rh}) = -27.31\sigma_p - 835.9$ ($R^2 = 0.9603$; $R = 0.980$).

σ_p-constants and Tolman basicity parameters, clearly the variation in δ(^{103}Rh) reflects the eq–eq/eq–ax ratio of these complexes.

The existence of a correlation between ligand basicity, the ratio of trigonal bipyramidal isomers and δ(^{103}Rh) indicates the significance of transition metal NMR for elucidation of structures of coordination compounds, especially in cases where NMR of more common nuclei give no or only partial answers. Work is currently in progress to further substantiate this possible application.

3. Experimental

3.1. Synthesis

Compounds $HRh(CO)_2$(diphosphine) have been synthesized in situ by reacting an appropriate amount of the diphosphine with $Rh(acac)(CO)_2$ in benzene-d_6 under nitrogen [19]. As an example, for the preparation of **4**, an amount of 0.0275 g (0.05 mmol) of the diphosphine thixantphos was added to a deuterobenzene solution of 0.0123 g of $Rh(acac)(CO)_2$. About 0.4 ml of the resulting solution was transferred into a 5 mm titanium–sapphire high-pressure NMR assembly [20,23], which was subsequently pressurized to 20 bar with synthesis gas (H_2:CO, 1:1) at 20°C.

Caution. High pressure experiments require adequate safety measures to be taken by the experimentalist [23]. The tube was heated at 80°C overnight and subsequently routine ^{1}H- and ^{31}P-NMR spectra were recorded prior to the ^{103}Rh-NMR measurements at 25°C. For variable temperature experiments, toluene-d_8 or thf-d_8 were employed at 80 and 50°C, respectively.

3.2. ^{103}Rh-NMR measurements

All ^{103}Rh-NMR spectra were measured on a 300 MHz Bruker Avance DRX spectrometer, δ values are in ppm referenced to $\Xi = 3.16$ MHz with positive shifts to higher frequency. The HMQC experiments were carried out using a 5 mm triple-resonance inverse probe-head (^{1}H,^{31}P{BB(^{103}Rh–^{31}P)}) with a z-gradient coil (90° $^1\text{H} = 7.2$ µs, 90° $^{31}\text{P} = 10.5$ µs, 90° $^{103}\text{Rh} = 19.5$ µs (300 W decoupler)) using for almost all compounds both of the sequences ^{1}H-{^{103}Rh}HMQC (see A) and ^{31}P-{^{103}Rh,^{1}H}HMQC (see B). (A) ^{1}H-{^{103}Rh}HMQC [2]. D1-90°(^{1}H)-1/2J-90°(^{103}Rh)-t_1/2-180°(^{1}H)-t_1/2-90°(^{103}Rh)-1/2J-ACQ. Acquisition parameters: $D1 = 1$ s, $1/2J = 0.1$–0.03 s, eight scans were acquired per t_1-increment, block size 2048 × 64 points for the first experiment and 2048 × 256 points for the high resolution experiment. For the first measurement a range of 10 ppm for ^{1}H and 10 000 ppm for ^{103}Rh (range from ca. −2000 to ca. 8000 ppm) was taken. Four-fold forward linear prediction [35] in F1, followed by one zerofill resulted in a resolution of 200 Hz per point in the ^{103}Rh domain. For the high resolution measurement the ^{103}Rh decoupler was set almost on resonance, spectral width in F1 was 3000 Hz. The resolution in the ^{103}Rh domain was, after linear prediction and zerofill as above, better than 3 Hz per point. Acquisition time 2.5–8 h (depending on the concentration) for the high resolution experiment. (B) ^{31}P-{^{103}Rh,^{1}H}HMQC [5]. Decoupling ^{1}H-D1-90°(^{31}P)-1/2J-90°(^{103}Rh)-t_1/2-180°(^{31}P)-t_1/2-90°(^{103}Rh)-1/2J-ACQ-decoupler off. Acquisition parameters: $D1 = 3$ s, $1/2J = 0.004$ s; 16–32 scans were acquired per t_1-increment, block size 2048 × 64 points for the first experiment and 2048 × 256 points for the high resolution experiment. For the first measurement a range of 50 ppm for ^{31}P and 10 000 ppm for ^{103}Rh (−2000↔8000 ppm) was taken, resulting in a resolution of 200 Hz per point after forward linear prediction and zerofill (see above). For the high resolution measurement the ^{103}Rh decoupler was set almost on resonance, spectral width in F1 3000 Hz. The resolution in the ^{103}Rh domain was, after linear prediction and zerofill as above, better than 3 Hz per point. Acquisition time 4–16 h (depending on the concentration) for the high resolution experiment.

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